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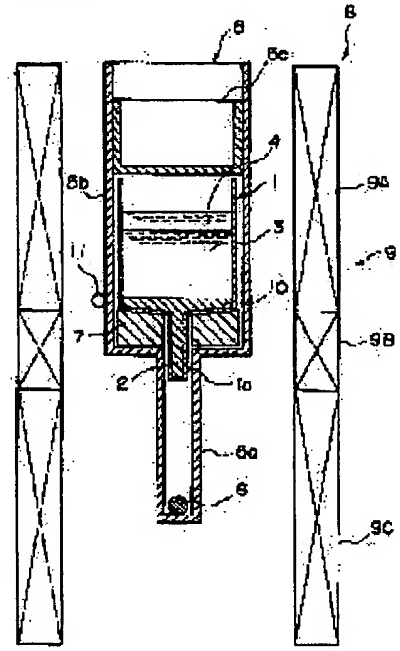
(54) PRODUCTION OF COMPOUND SEMICONDUCTOR SINGLE CRYSTAL

(57)Abstract:
PROBLEM TO BE SOLVED: To produce compound semiconductor single crystals, especially compound semiconductor single crystals of zinc-blend structure such as GaAs and InP, by a VGF method or VB method in high yield by preventing generation of twin crystals at a crystal diameter increasing part.
SOLUTION: In production of the compound semiconductor single crystals, a crucible 1, which has a seed crystal setting part 1a at a center of a bottom part, a bottom surface of which inclines with a prescribed angle α of 80°≤ and <90° to a vertical direction so that the botton surface gradually is lowered toward its center and in which a corner part between the bottom surface and a flank has radius of curvature of 0≤ to ≤10mm, is used. At the time of executing crystal growing by the VGF method (vertical gradient freezing method) or VB method (vertical Bridman method), the temp. gradient of a molten raw material 3 at least at an inclined bottom part of the crucible 1, that is, in a region from a solid-liquid boundary between the seed crystal 2 and the molten raw material 3 at a point of crystal growing starting to beginning of growing of a straight cylindrical part of the crystals, is set to 1≤ to <5° C/cm, preferably 2≤ to ≤4.5° C/cm and more preferably 3≤ to ≤4° C/cm.



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CLAIMS

[Claim(s)]
[Claim 1] Seed crystal is installed in the center of a pars basilaris ossis occipitalis at these seed crystal installation circles of a crucible that have the installation section of seed crystal. After putting in the raw material and encapsulant of a compound semiconductor in this crucible and enclosing the crucible in a tight container, Install this tight container in the heating furnace of a vertical mold, and heating fusion of said raw material and said encapsulant is carried out at a heater. In growing up the single crystal of a compound semiconductor by cooling the obtained raw material melt gradually from the bottom, and making it solidify toward the upper part from said seed crystal as said crucible While using the crucible which made predetermined 80-degree or more include angle of less than 90 degrees, and inclined to the perpendicular direction so that the base may become low gradually toward the core The manufacture approach of the compound semiconductor single crystal characterized by controlling the temperature gradient of the crystal growth direction of the inclined crucible bottom part to become in cm and 1 degrees C/cm or more less than 5 degrees C /at least at the time of crystal growth.
[Claim 2] The radius of curvature of the boundary part of the base of said crucible and a side face is the manufacture approach of the compound semiconductor single crystal according to claim 1 characterized by 0mm or more being 10mm or less.
[Claim 3] It is the manufacture approach of the compound semiconductor single crystal according to claim 1 or 2 characterized by controlling the temperature gradient of the crystal growth direction of said crucible bottom part preferably to be set to cm in 2 degrees C/cm or more 4.5 degrees C /or less.
[Claim 4] It is the manufacture approach of the compound semiconductor single crystal according to claim 1 or 2 characterized by controlling the temperature gradient of the crystal growth direction of said crucible bottom part more preferably to be set to cm in 3 degrees C/cm or more 4 degrees C /or less.
[Claim 5] The manufacture approach of the compound semiconductor single crystal according to claim 1, 2, 3, or 4 characterized by growing up the compound semiconductor single crystal of zincblende structure.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] perpendicular gradient freezing (hereafter referred to as VGF.) which this invention cools [freezing] the raw material melt of a compound semiconductor, concerning the manufacture approach of a compound semiconductor single crystal, and grows up a single crystal perpendicularly -- law and perpendicular Bridgman (hereafter referred to as VB.) -- it applies to law and is related with a useful technique.
[0002]
[Description of the Prior Art] general -- a compound semiconductor single crystal ingot -- manufacturing -- hitting -- liquid closure CHOKURARU skiing (LEC) -- law or level Bridgman (HB) -- law is used industrially. Since the liquid encapsulant (B-2 03) with which a wafer with a circular cross-section configuration is obtained with the diameter of macrostomia is used for an LEC method, while there are the advantages, -- the crystal of a high grade is obtained --, since the temperature gradient of the crystal growth direction is large, the dislocation density under crystal becomes high and there is demerit in which the electric property of electron devices, such as FET (field-effect transistor) produced using the crystal, will deteriorate. On the other hand, since the temperature gradient of the crystal growth direction is small, while there is the advantage in which the crystal of low dislocation density is obtained, in order to make the HB method solidify the raw material melt of a compound semiconductor within a crucible, the cross-section configuration of a wafer where diameter[of macrostomia]-izing is difficult and was obtained has the demerit of becoming a boiled-fish-paste form in it.
[0003] then -- as the single crystal manufacture approach of having each advantage of an LEC method and the HB method -- perpendicular gradient freezing (VGF) -- law and perpendicular Bridgman (VB) -- law is proposed. Since these VGF(s) method and a VB method have the small temperature gradient of the crystal growth direction where a circular wafer is obtained in order to use ****** of a cylindrical shape, they have the advantage in which the crystal of low dislocation density is obtained easily. However, in the VGF method and a VB method, it is [with the slight influential temperature fluctuation in a furnace] easy the irregularity of ****** been and stretched and the effect of a foreign matter, and there is a fault of being easy to generate twin crystal and polycrystal.
[0004] About the effect of the temperature fluctuation in a furnace, development of a temperature control technique in recent years has been solved among those faults. Moreover, it can prevent now by use of liquid encapsulant (B-2 O3) also about generating of the polycrystal from a crucible wall.
[0005] However, the still effective preventive measure is not proposed to generating of twin crystal. Especially, the probability of twin crystal generating in a crystal increase diameter until it results [from a crystal training start point] in the body section is high, and has become the main causes of reducing the yield of single crystal manufacture from the body section of a crystal.
[0006] when raising the compound semiconductor single crystal of zincblende structure like GaAs, InP, or GaP using seed crystal, it results from seed crystal to the body section -- an increase -- a diameter -- between an include angle and the probability of occurrence of twin crystal, it turns out that there is close relation. namely, an increase when raising the crystal of bearing (100) -- a diameter -- a facet (111) side appears and twin crystal is generated from this facet side. The experiment whose this invention persons performed this is also checked. That is, when this invention persons performed crystal training, as the crystal which twin crystal generated, all twin crystal was generated along with facet growth.
[0007] (111) A facet makes bearing (100) and the include angle of 54.7 degrees. therefore, an increase in order to prevent a facet (111) side appearing generally -- a diameter -- the include angle is made into [90-degree - 54.7 or less "], i.e., 35.3 degrees. however -- an increase -- a diameter -- the increase of a crystal acquired when the include angle was made small -- a diameter -- it will become long, the yield of a wafer falls and productivity is bad. then -- an increase -- a diameter -- although the attempt in which an include angle is made into 40 degrees - about 50 degrees is also made, effectiveness sufficient in respect of generating suppression of twin crystal is not acquired.
[0008] moreover -- JP,5-194073,A -- an increase -- a diameter -- while using the crucible [like] with which an include angle becomes 80 degrees - 100 degrees -- the field near the seed crystal -- local -- a supercooling condition -- carrying out -- abbreviation -- after growing up a crystal in the level direction and making it grow up so that it may crystallize upper convex one further, the crystal manufacture approach of

cooling under a 5 degrees C/cm - 15 degrees C [/cm] temperature gradient, and having made it solidify raw material melt is indicated. In order to cool raw material melt, holding a 5 degrees C/cm - 15 degrees C [/cm] temperature gradient, piping for cooling media is prepared in a heat sink, and he pours the medium for cooling and is trying to raise the heat dissipation nature of a heat sink into that piping by this manufacture approach. In addition, according to JP,5-194073,A, a temperature gradient cannot control the temperature distribution of melt easily so that the isothermal surface of raw material melt becomes convex voice by cm in less than 5 degrees C /at a melt side, and it is supposed that a single crystal cannot grow it easily. Moreover, if a temperature gradient exceeds cm in 15 degrees C /, it will solidify rapidly, and it is supposed that a dendrite arises and it will polycrystal-be easy toize.

[0009]

[Problem(s) to be Solved by the Invention] However, it was shown clearly by this invention persons for there to be the following problems in the crystal manufacture approach indicated by above-mentioned JP,5-194073,A. That is, in order to control the temperature distribution of melt by JP,5-194073,A so that the isothermal surface of raw material melt becomes convex voice at a melt side, it is supposed that the temperature gradient at the time of cooling must be carried out [cm] in 5 degrees C /or more. However, temperature fluctuation according [a temperature gradient] to the convection current in raw material melt in 5 degrees C/cm or more does not become small enough, but it is easy to generate twin crystal and polycrystal. That is, generating of twin crystal or polycrystal cannot be controlled to extent which can fully be satisfied. Moreover, in order to install piping for cooling media in a heat sink, there was also a fault that great cost started.

[0010] this invention was made in order to solve the above-mentioned trouble, and twin crystal generates it in a crystal increase diameter -- protecting -- the high yield -- a compound semiconductor single crystal especially GaAs, InP, etc. -- like -- the compound semiconductor single crystal of zincblende structure -- VGF -- it aims at offering the single crystal manufacture approach which can be manufactured by law or the VB method.

[0011]

[Means for Solving the Problem] in order to attain the above-mentioned purpose -- this invention persons -- abbreviation -- the probability of occurrence of twin crystal is high by performing crystal growth using the crucible of a flat base configuration -- an increase -- a diameter -- I thought that a crystal could be grown up, without forming. Moreover, by growing up a crystal in the shape of flatness from raw material melt, without making it grow up to be upper convex, the temperature gradient in raw material melt was made [cm] in less than 5 degrees C /, and I thought that temperature fluctuation could be made small. Furthermore, if the radius of curvature of the part which shifts to the body section of a training crystal from a crystal shoulder was large, since the number of facets which the time amount which the crystal growth of the part takes becomes long, and is generated would also increase, therefore it would become easy to generate twin crystal, I thought it effective to make into the value of predetermined within the limits the radius of curvature of the part which shifts to the body section from a crystal shoulder.

[0012] This invention was made based on the above-mentioned view, and seed crystal is installed in these seed crystal installation circles of a crucible that have the installation section of seed crystal in the center of a pars basilaris ossis occipitalis. After putting in the raw material and encapsulant of a compound semiconductor in this crucible and enclosing the crucible in a tight container, Install this tight container in the heating furnace of a vertical mold, and heating fusion of said raw material and said encapsulant is carried out at a heater. In growing up the single crystal of a compound semiconductor, especially the compound semiconductor of zincblende structure by cooling the obtained raw material melt gradually from the bottom, and making it solidify toward the upper part from said seed crystal as said crucible While using the crucible which made predetermined 80-degree or more include angle of less than 90 degrees, and inclined to the perpendicular direction so that the base may become low gradually toward the core At the time of crystal growth, at least, the temperature gradient of the crystal growth direction of the inclined crucible bottom part is controlled so that 1 degrees C/cm or more less than 5 degrees C /2 degrees C/cm or more 4.5 degrees C [/] or less become more preferably cm cm in cm and 3 degrees C/cm or more 4 degrees C /or less. Moreover, this invention sets the radius of curvature of the corner equivalent to the shift part from a crystal shoulder to the body section of the boundary part of the base of said crucible, and a side face, i.e., a training crystal, to 0mm or more 10mm or less.

[0013] the increase of it -- a diameter -- a crystal grows in the shape of flatness from raw material melt, without forming. Moreover, since the temperature gradient at the time of cooling raw material melt and making it solidify is small, temperature fluctuation becomes small. Moreover, the crystal growth time amount of the shift part from a crystal shoulder to the body section of a training crystal becomes short, generating of a facet is controlled, and generating of twin crystal is prevented. As a result of measuring temperature fluctuation with the thermocouple formed in contact with the outside of the part corresponding to the crucible bottom of the tight container which enclosed a raw material and encapsulant according to research of this invention persons, it is suitable for temperature fluctuation that it is **0.1 degrees C or less.

[0014]

[Embodiment of the Invention] The crucible used for operation of this invention is shown in drawing 1 . moreover -- drawing 2 -- this invention -- VGF -- the outline of the crystal growth furnace used when it

applies to law is shown.
[0015] By the single crystal manufacture approach concerning this invention, as shown in drawing 1 , seed crystal installation section 1a is prepared in the center of a pars basilaris ossis occipitalis of a crucible 1, and the crucible 1 which is formed and becomes so that base 1b of a crucible 1 may become low gradually toward the core, and the predetermined 80-degree or more include angle alpha of less than 90 degrees may be made and it may incline to a perpendicular direction is used. the reason the include angle alpha of base 1a of a crucible 1 is 80 degrees or more less than 90 degrees -- less than 80 degrees -- an increase -- a diameter -- a part -- since it is easy to attach a temperature gradient, temperature fluctuation becomes large as a result and twin crystal is generated -- it is -- 90 degrees or more -- an increase -- a diameter -- a part -- it is because other grain boundaries grow and it becomes polycrystal.
[0016] Moreover, the radius of curvature of 1d of corners by which a crucible 1 is equivalent to the shift part from a crystal shoulder to the body section of the boundary part of the base 1b and side-face 1c, i.e., a training crystal, is 0mm or more 10mm or less. The reason the radius of curvature of 1d of corners between base 1b and side-face 1c of a crucible 1 is 0mm or more 10mm or less is the probability of occurrence of twin crystal increases and not desirable according to this invention persons' examination result, if the radius of curvature is larger than 10mm. Moreover, about the minimum of the radius of curvature, since generating of a facet can be prevented so that radius of curvature is small, 1d of radius of curvatures of 0mm, i.e., a corner, does not need to be a curved surface.
[0017] And as shown in drawing 2 , seed crystal 2 is put in in seed crystal installation section 1a of a crucible 1, and the raw material 3 and encapsulant 4 of a compound semiconductor are put in in a crucible 1. The element 6 for vapor pressure control is put in in vapor pressure control-section (reservoir) 5a of a tight container 5, the crucible 1 is further installed on the susceptor 7 in crystal training section 5b of a tight container 5, evacuation of the inside of a tight container 5 is carried out, and it closes by cap 5c. The element 6 for vapor pressure control is the simple substance or compound which consists of an element which is easy to volatilize among the configuration elements of the single crystal to grow up.
[0018] The tight container 5 is installed in the predetermined location in the vertical mold heating furnace 8, it heats at a heater 9, and a raw material 3 and encapsulant 4 are dissolved. Although not limited especially, it is good to use the heater of the three-step configuration of the shape of a cylinder which considers as a heater 9, for example, consists of heater 9for the crystal training sections A, heater 9for the seed crystal sections B, and heater 9C for vapor pressure control sections at least.
[0019] Each output of each [these] heaters 9A, 9B, and 9C is adjusted, and a single crystal 10 is grown up toward the upper part by cooling raw material melt 3 from the lower part to the temperature below the melting point gradually, maintaining a predetermined temperature gradient which serves as an elevated temperature from a seed crystal 2 side gradually toward the upper part of raw material melt 3. The vapor pressure in a tight container 5 is maintained at a suitable pressure by output adjustment of heater 9C for vapor pressure control sections in that case.
[0020] The bottom part toward which the crucible 1 inclined at least about the temperature gradient at the time of cooling here, Namely, a field until training of the body section of a crystal is started from the solid-liquid interface of the seed crystal 2 at the crystal training initiation time, and raw material melt 3 (drawing 1 reference) It is appropriate that 1 degrees C/cm or more less than 5 degrees C /of 2 degrees C/cm or more 4.5 degrees C /or less of temperature gradients in the field of D of this drawing are more preferably set to cm cm cm in 3 degrees C/cm or more 4 degrees C /or less. The reason is that a temperature gradient becomes easy to be influenced of ambient temperature by cm in less than 1 degree C /, and is because temperature fluctuation becomes large by cm in 5 degrees C /or more. Moreover, there is an advantage that a proper training rate and the rate of single-crystal-izing will be obtained if a temperature gradient is 2 degrees C/cm - 4.5 degrees C/cm, and it is more desirable if a temperature gradient is 3 degrees C/cm - 4 degrees C/cm further.
[0021] Moreover, it checks that form a thermocouple 11 in contact with the outside of the part corresponding to the crucible bottom of a tight container 5, measure temperature fluctuation with the thermocouple 11, and temperature fluctuation has magnitude of predetermined within the limits. In addition, you may make it adjust the output of each heaters 9A, 9B, and 9C so that the measured temperature fluctuation may become the magnitude of predetermined within the limits. About the tolerance of temperature fluctuation, it asks by preliminary experiment etc. As a result of conducting preliminary experiment as the configuration shown in drawing 2 is also, it turned out that the tolerance of temperature fluctuation is **0.1 degrees C or less. The reason is that it will become easy to generate twin crystal and polycrystal if temperature fluctuation deviates from the tolerance.
[0022] While according to the above-mentioned operation gestalt making the predetermined 80-degree or more include angle alpha of less than 90 degrees and inclining to a perpendicular direction so that base 1b of a crucible 1 may become low gradually toward the core The radius of curvature of 1d of corners between base 1b and side-face 1c uses the 0mm or more crucible [like] 1 it is [crucible] 10mm or less. The temperature gradient of the crystal growth direction of the inclined crucible bottom part (said field D) at least 1 degrees C/cm or more less than 5 degrees C/cm while controlling preferably so that 2 degrees C/cm or more 4.5 degrees C /or less are more preferably set to cm cm in 3 degrees C/cm or more 4 degrees C /or less -- raw material melt 3 -- gradually -- cooling -- VGF, since it was made to grow up a compound semiconductor

single crystal by law the increase of crystal 10 -- a diameter -- after a crystal 10 grows in the shape of flatness along with base 1b of a crucible 1 from raw material melt 3 first, without being formed, while the solid-liquid interface had made the shape of flatness, a crystal 10 grows toward the upper part further. Therefore, since there is no crystal increase diameter, the yield of the wafer from the obtained single crystal ingot is high, and productivity is good. moreover, the temperature gradient at the time of solidification of raw material melt 3 -- small -- a thing with small temperature fluctuation -- in addition, since generating of the facet at the time of carrying out body section HE shift from a crystal shoulder is controlled, generating of twin crystal and polycrystal is controlled and a single crystal is obtained by the high yield.
[0023] Furthermore, the single crystal of high quality without twin crystal or polycrystal is obtained by the high yield, without causing the increment in cost, since a heating furnace 8 has unnecessary piping for cooling media etc., and the conventional heating furnace can be used as it is.
[0024] in addition, the gestalt of the above-mentioned implementation -- setting -- this invention -- VGF -- although the case where it applied to law was explained, this invention is applicable also to a VB method.
[0025]
[Example] The place by which gives an example and the example of a comparison to below, and it is characterized [of this invention] is clarified. In addition, this invention is not limited at all by each following example.
[0026] (Example 1) As a crucible, the diameter used the crucible 1 made from pBN of the configuration which thickness shows to drawing 1 which is 3mm by about 3 inches. Moreover, the include angle alpha which base 1b of an end crater 1 which receives perpendicularly makes was made into 87 degrees C. Moreover, the radius of curvature of 1d of corners between base 1b and side-face 1c of a crucible 1 was set to 4mm.
[0027] The seed crystal 2 set to seed crystal installation section 1a of a crucible 1 from a GaAs single crystal is put in, and it is B-2 O3 of optimum dose as about 3kg GaAs polycrystal and encapsulant 4 as a raw material 3 in a crucible 1 further. It put in. Then, after installing the crucible 1 into which 8g arsenic was put into vapor pressure control-section 5a of the quartz ampul which is a tight container 5 as an element 6 for vapor pressure control, and a raw material 3 and encapsulant 4 were put on the susceptor 7 in quartz ampul, the vacuum lock was carried out by cap 5c. And the tight container 5 was installed in the vertical mold heating furnace 8 of a three-step heater configuration, as shown in drawing 2 R> 2. In addition, Ga and As are put in in a crucible 1 and you may make it make them compound directly instead of using GaAs polycrystal as a raw material 3.
[0028] While heating the end crater 1 boiled so that it may become the upper limit of seed crystal 2, and the temperature whose raw material 3 is 1238 degrees C - 1255 degrees C and dissolving a raw material 3 and encapsulant 4 by heater 9for the crystal training sections A, and heater 9B for the seed crystal sections, it heated so that it might become 605 degrees C about vapor pressure control-section 5a by heater 9C for vapor pressure control sections. Moreover, it was made for the temperature gradient in the field D until training of the body section of a crystal is started from the solid-liquid interface of the bottom part, i.e., the seed crystal 2 at the crystal training initiation time and raw material melt 3, toward which the crucible 1 inclined to be set to cm in 3.5 degrees C /. The temperature fluctuation measured with the thermocouple 11 at this time was **0.06 degrees C.
[0029] In this condition, the laying temperature of a heating furnace 8 was lowered continuously, and training of a crystal was started so that the training rate of a crystal might be set to per hour 2mm. When about 30 hours had passed since crystal training initiation, raw material melt 3 was solidified altogether. Then, the heating furnace 8 whole was cooled at the temperature fall rate of 100 degrees C/h, when it got cold to near the room temperature, the tight container 5 was taken out from the inside of a heating furnace 8, the tight container 5 was broken and the crystal was taken out. The obtained crystal was a GaAs single crystal of crystal orientation (100) with an overall length of about 12cm for the diameter of about 3 inches, and when the crystallinity was investigated, twin crystal or polycrystal were not generated at all. Moreover, this single crystal ingot was cut and dislocation density was two or less [1000cm -] in every field of a crystal at ****** and the time about dislocation density.
[0030] When single crystal growth of GaAs was performed 20 times on the same conditions as the above-mentioned example, about 18 times, the single crystal without twin crystal or polycrystal was obtained among those.
[0031] (Example 2) Except having set the radius of curvature of 1d of corners between base 1b and side-face 1c of a crucible 1 to 10mm, it is the same conditions as the above-mentioned example 1, and single crystal growth of GaAs was performed 5 times. Consequently, about 4 times of crystal growth, the single crystal without twin crystal or polycrystal was obtained. the above-mentioned example 1 -- radius of curvature -- a few -- large -- having carried out (4mm having been set to 10mm) -- time -- a single crystal -- although the yield fell for a while, the yield was better than the example of a comparison mentioned later.
[0032] in addition, except for GaAs -- the compound semiconductor of zincblende structures, such as InP and GaP, -- VGF -- this invention is effective also when manufacturing by law or the VB method.
[0033] (Example 1 of a comparison) The GaAs single crystal was manufactured like the above-mentioned example 1 using a crucible [as / whose include angle alpha which the base of the end crater which receives perpendicularly makes is 30 degrees] made from pBN. In addition, conditions other than the include angle alpha of a crucible bottom were the same as the above-mentioned example 1. Since twin crystal was

generated in the crystal increase diameter until it results [from seed crystal] in the obtained crystal at the body section and bearing had changed, it was impossible to have used the crystal as a crystal for semi-conductor substrates. When single crystal growth of GaAs was performed 5 times on the same conditions, although two in the obtained crystal of five were a single crystal, twin crystal was generated into the crystal of three and they were unusable.

[0034] (Example 2 of a comparison) The same crucible 1 as the above-mentioned example 1 was used, the temperature gradient of the bottom part (said field D) toward which the crucible 1 inclined was set [cm] up in 15 degrees C /, and the GaAs single crystal was manufactured like the above-mentioned example 1. In addition, conditions other than the temperature gradient of the bottom part of a crucible 1 were the same as the above-mentioned example. When the temperature fluctuation at the time of crystal growth was measured with the thermocouple 11, it was **0.3 degrees C. the obtained crystal -- the -- an increase -- a diameter -- it was impossible for polycrystal to have been generated and to have used the crystal as a crystal for semi-conductor substrates. When single crystal growth of GaAs was performed 5 times on the same conditions, it was one that it was a single crystal among the obtained crystals of five, polycrystal was generated into other crystals of four and it was unusable. Moreover, when the GaAs crystal of the single crystal obtained only one was cut and dislocation density was investigated, in every field of a crystal, dislocation density was over 5000cm-2.

[0035]

[Effect of the Invention] According to this invention, install seed crystal in the seed crystal installation circles of a crucible pars basilaris ossis occipitalis, and the raw material and encapsulant of a compound semiconductor are put in in the crucible. After enclosing the crucible in a tight container, install this tight container in the heating furnace of a vertical mold, and heating fusion of said raw material and said encapsulant is carried out at a heater. In growing up the single crystal of a compound semiconductor by cooling the obtained raw material melt gradually from the bottom, and making it solidify toward the upper part from said seed crystal as said crucible While using the crucible which made predetermined 80-degree or more include angle of less than 90 degrees, and inclined to the perpendicular direction so that the base may become low gradually toward the core In order to control the temperature gradient of the crystal growth direction of the inclined crucible bottom part at least to become in cm and 1 degrees C/cm or more less than 5 degrees C /and to perform crystal growth at the time of crystal growth, the increase of a crystal -- a diameter -- since training of the body section is immediately started after crystal training initiation, without being formed, the yield of the wafer from the obtained single crystal ingot is high, and productivity is good. Moreover, the temperature gradient at the time of solidification of raw material melt is small, since temperature fluctuation is small, generating of twin crystal and polycrystal is controlled and a single crystal is obtained by the high yield. Furthermore, the single crystal of high quality is obtained by the high yield, without causing the increment in cost, since piping of a heating furnace for cooling media etc. is unnecessary, and the conventional heating furnace can be used as it is.

[0036] Moreover, according to this invention, since the radius of curvature of the boundary part of the base of said crucible and a side face is 0mm or more 10mm or less, the crystal growth time amount of the shift part from a crystal shoulder to the body section of a training crystal becomes short, generating of a facet is controlled, and generating of twin crystal is prevented.

[Translation done.]

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the sectional view of an example of the crucible used for operation of this invention.
[Drawing 2] It is the schematic diagram of the crystal growth furnace used when this invention is applied to the VGF method.
[Description of Notations]
1 Crucible
1a Seed crystal installation section
1b The base of a crucible
1c The side face of a crucible
1d Corner of a crucible
2 Seed Crystal
3 Raw Material
4 Encapsulant
5 Tight Container
8 Heating Furnace (Crystal Growth Furnace)
9 Heater
10 Single Crystal

[Translation done.]

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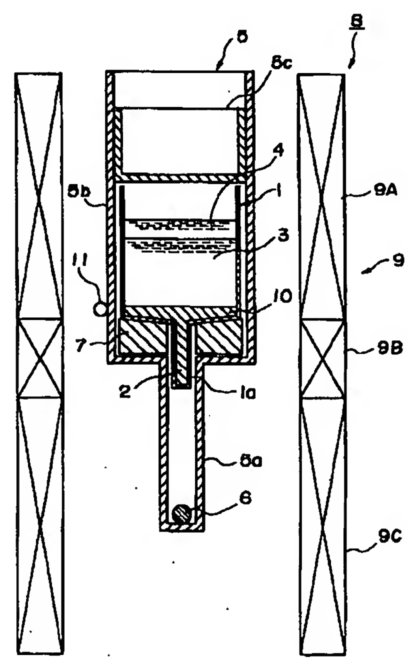
(54)【発明の名称】　化合物半導体単結晶の製造方法

(57)【要約】

【課題】　結晶増径部にて双晶が発生するのを防いで高い歩留まりで化合物半導体単結晶、特にＧａＡｓやＩｎＰ等のように閃亜鉛鉱型構造の化合物半導体単結晶をＶＧＦ法やＶＢ法により製造する。

【解決手段】　底部中央に種結晶設置部１ａを有し、かつ底面１ｂがその中心に向かって徐々に低くなるように垂直方向に対して８０°以上９０°未満の所定角度αで傾斜しているとともに、底面１ｂと側面１ｃとの間の角部１ｄの曲率半径が０mm以上１０mm以下であるようなるつぼ１を用いる。ＶＧＦ法やＶＢ法により結晶育成を行う際の、原料融液３の温度勾配を、少なくともるつぼ１の傾斜した底部分、すなわち結晶育成開始時点の種結晶２と原料融液３との固液界面から結晶の直胴部の育成が開始されるまでの領域における温度勾配が１℃／cm以上５℃／cm未満、好ましくは２℃／cm以上４．５℃／cm以下、より好ましくは３℃／cm以上４℃／cm以下となるようにする。



【特許請求の範囲】

【請求項１】　底部中央に種結晶の設置部を有するるつぼの該種結晶設置部内に種結晶を設置し、該るつぼ内に化合物半導体の原料及び封止剤を入れ、そのるつぼを気密容器内に封入した後、該気密容器を縦型の加熱炉内に設置して前記原料及び前記封止剤をヒータにより加熱融解し、得られた原料融液を下側から徐々に冷却して前記種結晶から上方に向かって固化させることにより化合物半導体の単結晶を成長させるにあたって、前記るつぼとして、その底面がその中心に向かって徐々に低くなるように垂直方向に対して８０°以上９０°未満の所定角度をなして傾斜したるつぼを用いるとともに、結晶成長時に少なくともその傾斜したるつぼ底部分の結晶成長方向の温度勾配を１℃／cm以上５℃／cm未満となるように制御することを特徴とする化合物半導体単結晶の製造方法。

【請求項２】　前記るつぼの底面と側面との境界部分の曲率半径は０mm以上１０mm以下であることを特徴とする請求項１記載の化合物半導体単結晶の製造方法。

【請求項３】　好ましくは、前記るつぼ底部分の結晶成長方向の温度勾配を２℃／cm以上４．５℃／cm以下となるように制御することを特徴とする請求項１または２記載の化合物半導体単結晶の製造方法。

【請求項４】　より好ましくは、前記るつぼ底部分の結晶成長方向の温度勾配を３℃／cm以上４℃／cm以下となるように制御することを特徴とする請求項１または２記載の化合物半導体単結晶の製造方法。

【請求項５】　閃亜鉛鉱型構造の化合物半導体単結晶を成長させることを特徴とする請求項１、２、３または４記載の化合物半導体単結晶の製造方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、化合物半導体単結晶の製造方法に関し、例えば化合物半導体の原料融液を冷却して垂直方向に単結晶を成長させる垂直グラジェントフリージング（以下、ＶＧＦとする。）法や垂直ブリッジマン（以下、ＶＢとする。）法に適用して有用な技術に関する。

【０００２】

【従来の技術】一般に、化合物半導体単結晶インゴットを製造するにあたって、液体封止チョクラルスキー（ＬＥＣ）法もしくは水平ブリッジマン（ＨＢ）法が工業的に用いられている。ＬＥＣ法には、大口径で断面形状が円形のウエハーが得られる、液体封止剤（B_2O_3）を使用しているため高純度の結晶が得られるなどの長所がある反面、結晶成長方向の温度勾配が大きいため結晶中の転位密度が高くなり、その結晶を用いて作製したＦＥＴ（電界効果トランジスタ）等の電子デバイスの電気的な特性が劣化してしまうという短所がある。一方、ＨＢ法には、結晶成長方向の温度勾配が小さいため低転位密度の結晶が得られるという長所がある反面、るつぼ内で化合物半導体の原料融液を固化させるため大口径化が困難である、得られたウエハーの断面形状はかまぼこ形になってしまうなどの短所がある。

【０００３】そこで、ＬＥＣ法とＨＢ法のそれぞれの長所を併せ持つ単結晶製造方法として、垂直グラジェントフリージング（ＶＧＦ）法や垂直ブリッジマン（ＶＢ）法が提案されている。これらＶＧＦ法やＶＢ法は、円筒形のるつぼを使用するため円形のウエハーが得られる、結晶成長方向の温度勾配が小さいため低転位密度の結晶が容易に得られるという長所を有する。しかし、ＶＧＦ法及びＶＢ法においては、炉内のわずかな温度変動の影響あるいはるつぼ壁の凹凸や異物の影響を受けやすく、双晶や多結晶が発生しやすいという欠点がある。

【０００４】それらの欠点のうち、炉内の温度変動の影響については、近年の温調技術の発展により解消されてきている。また、るつぼ壁からの多結晶の発生についても、液体封止剤（B_2O_3）の使用により防止できるようになった。

【０００５】しかしながら、双晶の発生に対しては未だ有効な防止策は提案されていない。特に、結晶の直胴部よりも、結晶育成開始点から直胴部に至るまでの結晶増径部における双晶発生の確率が高く、単結晶製造の歩留りを低下させる主な原因となっている。

【０００６】ＧａＡｓやＩｎＰやＧａＰのような閃亜鉛鉱型構造の化合物半導体単結晶を種結晶を用いて育成する場合、種結晶から直胴部へ至る増径部の角度と双晶の発生確率との間には密接な関係があることがわかっている。すなわち、（１００）方位の結晶を育成する場合、増径部に（１１１）ファセット面が現れ、このファセット面から双晶が発生する。このことは、本発明者らの行った実験でも確認されている。すなわち、本発明者らが結晶育成を行ったところ、双晶の発生した結晶では、全ての双晶がファセット成長に沿って発生していた。

【０００７】（１１１）ファセットは（１００）方位と５４．７°の角度をなす。従って、一般には、（１１１）ファセット面が現れるのを防ぐために、増径部の角度を［９０°－５４．７°］すなわち３５．３°以下としている。しかし、増径部の角度を小さくすると、得られた結晶は増径部の長いものとなってしまい、ウエハーの収率が低下し生産性が悪い。そこで、増径部の角度を４０°～５０°程度にするという試みもなされているが、双晶の発生抑止という点では十分な効果が得られていない。

【０００８】また、特開平５－１９４０７３号公報には、増径部の角度が８０°～１００°となるようなるつぼを用いるとともに、種結晶近傍の領域を局所的に過冷却状態にして略水平な方向に結晶を成長させ、さらに結晶を上凸状をなすように成長させた後、原料融液を５℃／cm～１５℃／cmの温度勾配下で冷却して固化させるよ

うにした結晶製造方法が開示されている。この製造方法では、５℃／cm～１５℃／cmの温度勾配を保持しつつ原料融液を冷却するために、ヒートシンクに冷却媒体用の配管を設け、その配管内に冷却用媒体を流してヒートシンクの放熱性を高めるようにしている。なお、特開平５－１９４０７３号公報によれば、温度勾配が５℃／cm未満では、原料融液の等温面が融液側に凸状態になるように融液の温度分布を制御し難く、単結晶が成長し難いとされている。また、温度勾配が１５℃／cmを超えると急激に固化してしまい、デンドライトが生じて多結晶化しやすいとされている。

【０００９】

【発明が解決しようとする課題】しかしながら、上記特開平５－１９４０７３号公報に開示された結晶製造方法には、次のような問題があることが本発明者らにより明らかとされた。すなわち、特開平５－１９４０７３号では、原料融液の等温面が融液側に凸状態になるように融液の温度分布を制御するために、冷却時の温度勾配を５℃／cm以上にしなければならないとしている。しかし、温度勾配が５℃／cm以上では原料融液中の対流による温度ゆらぎは十分に小さくならず、双晶や多結晶が発生しやすい。すなわち、十分に満足できる程度に双晶や多結晶の発生を抑制することができない。また、ヒートシンク内に冷却媒体用の配管を設置するため、多大なコストがかかるという欠点もあった。

【００１０】本発明は、上記問題点を解決するためになされたもので、結晶増径部にて双晶が発生するのを防いで高い歩留まりで化合物半導体単結晶、特にＧａＡｓやＩｎＰ等のように閃亜鉛鉱型構造の化合物半導体単結晶をＶＧＦ法やＶＢ法により製造することができる単結晶製造方法を提供することを目的とする。

【００１１】

【課題を解決するための手段】上記目的を達成するために、本発明者らは、略平坦な底面形状のるつぼを用いて結晶成長を行うことによって、双晶の発生確率の高い増径部を形成することなく結晶を成長させることができると考えた。また、原料融液から結晶を上凸状に成長させずに平坦状に成長させることにより、原料融液中の温度勾配を５℃／cm未満にでき、温度ゆらぎを小さくすることができると考えた。さらに、育成結晶の、結晶肩部から直胴部へ移行する部分の曲率半径が大きいと、その部分の結晶成長に要する時間が長くなって発生するファセット数も増え、従って双晶が発生し易くなるので、結晶肩部から直胴部へ移行する部分の曲率半径を所定範囲内の値とすることが有効であると考えた。

【００１２】本発明は、上記着眼に基づきなされたもので、底部中央に種結晶の設置部を有するるつぼの該種結晶設置部内に種結晶を設置し、該るつぼ内に化合物半導体の原料及び封止剤を入れ、そのるつぼを気密容器内に封入した後、該気密容器を縦型の加熱炉内に設置して前記原料及び前記封止剤をヒータにより加熱融解し、得られた原料融液を下側から徐々に冷却して前記種結晶から上方に向かって固化させることにより化合物半導体、特に閃亜鉛鉱型構造の化合物半導体の単結晶を成長させるにあたって、前記るつぼとして、その底面がその中心に向かって徐々に低くなるように垂直方向に対して８０°以上９０°未満の所定角度をなして傾斜したるつぼを用いるとともに、結晶成長時に少なくともその傾斜したるつぼ底部分の結晶成長方向の温度勾配を１℃／cm以上５℃／cm未満、好ましくは２℃／cm以上４．５℃／cm以下、より好ましくは３℃／cm以上４℃／cm以下となるように制御するものである。また、本発明は、前記るつぼの底面と側面との境界部分、すなわち育成結晶の、結晶肩部から直胴部への移行部分に相当する角部の曲率半径を０mm以上１０mm以下とするものである。

【００１３】それによって、増径部を形成することなく原料融液から結晶が平坦状に成長する。また、原料融液を冷却して固化させる際の温度勾配が小さいため、温度ゆらぎが小さくなる。また、育成結晶の、結晶肩部から直胴部への移行部分の結晶成長時間が短くなり、ファセットの発生が抑制され、双晶の発生が防止される。本発明者らの研究によれば、原料及び封止剤を封入した気密容器の、るつぼ底に対応する箇所の外側に接して設けた熱電対により温度ゆらぎを測定した結果、温度ゆらぎは±０．１℃以下であるのが適当である。

【００１４】

【発明の実施の形態】図１には、本発明の実施に使用されるるつぼが示されている。また、図２には、本発明をＶＧＦ法に適用した際に使用される結晶成長炉の概略が示されている。

【００１５】本発明に係る単結晶製造方法では、図１に示すように、るつぼ１の底部中央に種結晶設置部１ａが設けられ、かつるつぼ１の底面１ｂがその中心に向かって徐々に低くなるように垂直方向に対して８０°以上９０°未満の所定角度αをなして傾斜するように形成されてなるるつぼ１を用いる。るつぼ１の底面１ａの角度αが８０°以上９０°未満である理由は、８０°未満では増径部分の温度勾配がつき易く、その結果温度ゆらぎが大きくなって、双晶が発生するためであり、９０°以上では増径部分で他粒界が成長し、多結晶となるからである。

【００１６】また、るつぼ１は、その底面１ｂと側面１ｃとの境界部分、すなわち育成結晶の、結晶肩部から直胴部への移行部分に相当する角部１ｄの曲率半径が０mm以上１０mm以下となっているものである。るつぼ１の、底面１ｂと側面１ｃとの間の角部１ｄの曲率半径が０mm以上１０mm以下である理由は、本発明者らの検討結果によれば、その曲率半径が１０mmよりも大きいと、双晶の発生確率が増大してしまい、好ましくないからである。また、その曲率半径の下限については、曲率半径が小さ

いほどファセットの発生を防止することができるので、曲率半径０mm、すなわち角部１ｄが曲面になっていなくてもよい。

【００１７】そして、図２に示すように、るつぼ１の種結晶設置部１ａ内に種結晶２を入れ、るつぼ１内に化合物半導体の原料３と封止剤４を入れる。気密容器５の蒸気圧制御部（リザーバ）５ａ内に蒸気圧制御用の元素６を入れ、さらに気密容器５の結晶育成部５ｂ内のサセプタ７上にそのるつぼ１を設置し、気密容器５内を真空排気してキャップ５ｃにより封止する。蒸気圧制御用元素６は、成長させる単結晶の構成元素のうち揮発し易い元素よりなる単体もしくは化合物である。

【００１８】その気密容器５を縦型加熱炉８内の所定位置に設置し、ヒータ９により加熱して原料３及び封止剤４を融解させる。特に限定しないが、ヒータ９として例えば少なくとも結晶育成部用ヒータ９Ａ、種結晶部用ヒータ９Ｂ及び蒸気圧制御部用ヒータ９Ｃからなる円筒状の３段構成のヒータを用いるとよい。

【００１９】それら各ヒータ９Ａ，９Ｂ，９Ｃの各出力を調整して、種結晶２側から原料融液３の上方に向かって徐々に高温となるような所定の温度勾配を維持しつつ徐々に原料融液３を下部から融点以下の温度に冷却することにより単結晶１０を上方に向かって成長させる。その際、気密容器５内の蒸気圧は、蒸気圧制御部用ヒータ９Ｃの出力調整により適当な圧力に保たれる。

【００２０】ここで、冷却時の温度勾配については、少なくともるつぼ１の傾斜した底部分、すなわち結晶育成開始時点の種結晶２と原料融液３との固液界面から結晶の直胴部の育成が開始されるまでの領域（図１参照、同図のＤの領域）における温度勾配が、１℃／cm以上５℃／cm未満、好ましくは２℃／cm以上４．５℃／cm以下、より好ましくは３℃／cm以上４℃／cm以下となるのが適当である。その理由は、温度勾配が１℃／cm未満では雰囲気温度の影響を受け易くなるからであり、５℃／cm以上では温度ゆらぎが大きくなるからである。また、温度勾配が２℃／cm～４．５℃／cmであれば、適正な育成速度と単結晶化率が得られるという利点が有り、さらに温度勾配が３℃／cm～４℃／cmであれば、より好ましい。

【００２１】また、気密容器５の、るつぼ底に対応する箇所の外側に熱電対１１を接して設け、その熱電対１１により温度ゆらぎを測定して温度ゆらぎが所定範囲内の大きさになっていることを確認する。なお、その測定した温度ゆらぎが所定範囲内の大きさになるように各ヒータ９Ａ，９Ｂ，９Ｃの出力を調整するようにしてもよい。温度ゆらぎの許容範囲については、予備実験等により求めておく。図２に示す構成でもって予備実験を行った結果、温度ゆらぎの許容範囲は±０．１℃以下であることがわかった。その理由は、温度ゆらぎがその許容範囲を逸脱すると、双晶や多結晶が発生しやすくなるからである。

【００２２】上記実施形態によれば、るつぼ１の底面１ｂがその中心に向かって徐々に低くなるように垂直方向に対して８０°以上９０°未満の所定角度αをなして傾斜しているとともに、底面１ｂと側面１ｃとの間の角部１ｄの曲率半径が０mm以上１０mm以下であるようなるつぼ１を用い、少なくともその傾斜したるつぼ底部分（前記領域Ｄ）の結晶成長方向の温度勾配が１℃／cm以上５℃／cm未満、好ましくは２℃／cm以上４．５℃／cm以下、より好ましくは３℃／cm以上４℃／cm以下となるように制御しながら原料融液３を徐々に冷却してＶＧＦ法により化合物半導体単結晶を成長させるようにしたため、結晶１０の増径部が形成されずにまず原料融液３から結晶１０がるつぼ１の底面１ｂに沿って平坦状に成長した後、固液界面が平坦状をなしたままさらに結晶１０が上方に向かって成長する。従って、結晶増径部がないため、得られた単結晶インゴットからのウエハーの収率が高く生産性がよい。また、原料融液３の固化時の温度勾配が小さく、温度ゆらぎが小さいのに加えて、結晶肩部から直胴部へ移行する際のファセットの発生が抑制されるので、双晶や多結晶の発生が抑制され、高い歩留まりで単結晶が得られる。

【００２３】さらに、加熱炉８は冷却媒体用の配管等が不要であるため、従来の加熱炉をそのまま使うことができるので、コストの増加を招くことなく、双晶や多結晶のない高品質の単結晶が高歩留まりで得られる。

【００２４】なお、上記実施の形態においては本発明をＶＧＦ法に適用した場合について説明したが、本発明はＶＢ法にも適用可能である。

【００２５】

【実施例】以下に、実施例及び比較例を挙げて本発明の特徴とするところを明らかとする。なお、本発明は、以下の各実施例により何ら限定されるものではない。

【００２６】（実施例１）るつぼとして、直径が約３インチで厚さが３mmの図１に示す形状のｐＢＮ製るつぼ１を用いた。また、垂直方向に対してるつぼ１の底面１ｂがなす角度αを８７℃とした。また、るつぼ１の、底面１ｂと側面１ｃとの間の角部１ｄの曲率半径を４mmとした。

【００２７】るつぼ１の種結晶設置部１ａにＧａＡｓ単結晶よりなる種結晶２を入れ、さらにるつぼ１内に原料３として約３kgのＧａＡｓ多結晶と封止剤４として適量のB_2O_3を入れた。続いて、気密容器５である石英アンプルの蒸気圧制御部５ａに蒸気圧制御用元素６として８ｇの砒素を入れ、原料３及び封止剤４を入れたるつぼ１を石英アンプル内のサセプタ７上に設置した後、キャップ５ｃにより真空封止した。そして、気密容器５を図２に示すように３段ヒータ構成の縦型加熱炉８内に設置した。なお、原料３としてＧａＡｓ多結晶を用いる代わりに、るつぼ１内にＧａとＡｓを入れてそれらを直接合成させるようにしてもよい。

【００２８】結晶育成部用ヒータ９Ａ及び種結晶部用ヒータ９Ｂにより、種結晶２の上端と原料３が１２３８℃～１２５５℃の温度となるようにるつぼ１を加熱して原料３及び封止剤４を融解させるとともに、蒸気圧制御部用ヒータ９Ｃにより蒸気圧制御部５ａを６０５℃となるように加熱した。また、るつぼ１の傾斜した底部分すなわち結晶育成開始時点の種結晶２と原料融液３との固液界面から結晶の直胴部の育成が開始されるまでの領域Ｄにおける温度勾配が３．５℃／cmとなるようにした。この時の熱電対１１により測定した温度ゆらぎは±０．０６℃であった。

【００２９】この状態で、結晶の育成速度が毎時２mmとなるように加熱炉８の設定温度を連続的に下げて結晶の育成を開始した。結晶育成開始から約３０時間経過した時点で原料融液３はすべて固化した。その後、加熱炉８全体を毎時１００℃の降温速度で冷却し、室温近くまで冷えた時点で加熱炉８内から気密容器５を取り出し、気密容器５を壊して結晶を取り出した。得られた結晶は直径約３インチで全長約１２cmの結晶方位（１００）のＧａＡｓ単結晶であり、その結晶性を調べたところ双晶や多結晶は全く発生していなかった。また、この単結晶インゴットを切断して転位密度を調べたところ、結晶のどの領域においても転位密度は１０００cm^{-2}以下であった。

【００３０】上記実施例と同一の条件でＧａＡｓの単結晶成長を２０回行ったところ、そのうち１８回については、双晶や多結晶のない単結晶が得られた。

【００３１】（実施例２）るつぼ１の、底面１ｂと側面１ｃとの間の角部１ｄの曲率半径を１０mmとした以外は、上記実施例１と同じ条件で、ＧａＡｓの単結晶成長を５回行った。その結果、４回の結晶成長については、双晶や多結晶のない単結晶が得られた。上記実施例１よりも曲率半径を少し大きくした（４mmを１０mmとした）ところ、単結晶の歩留りが少し低下したが、後述する比較例よりも歩留りが良かった。

【００３２】なお、ＧａＡｓ以外にもＩｎＰやＧａＰなどの閃亜鉛鉱型構造の化合物半導体をＶＧＦ法やＶＢ法により製造する場合にも本発明は有効である。

【００３３】（比較例１）垂直方向に対してるつぼの底面のなす角度αが３０°であるようなｐＢＮ製のるつぼを用い、上記実施例１と同様にして、ＧａＡｓ単結晶の製造を行った。なお、るつぼ底の角度α以外の条件は上記実施例１と同じであった。得られた結晶には種結晶から直胴部に至るまでの結晶増径部に双晶が発生しており、方位が変わってしまったため、その結晶を半導体基板用の結晶として使用することは不可能であった。同一の条件でＧａＡｓの単結晶成長を５回行ったところ、得られた５本の結晶のうち２本は単結晶であったが、３本の結晶には双晶が発生しており使用不可能であった。

【００３４】（比較例２）上記実施例１と同じるつぼ１を使用し、るつぼ１の傾斜した底部分（前記領域Ｄ）の温度勾配を１５℃／cmに設定して、上記実施例１と同様にしてＧａＡｓ単結晶の製造を行った。なお、るつぼ１の底部分の温度勾配以外の条件は上記実施例と同じであった。結晶成長時の温度ゆらぎを熱電対１１により測定したところ、±０．３℃であった。得られた結晶にはその増径部に多結晶が発生しており、その結晶を半導体基板用の結晶として使用することは不可能であった。同一の条件でＧａＡｓの単結晶成長を５回行ったところ、得られた５本の結晶のうち単結晶であったのは１本だけであり、他の４本の結晶には多結晶が発生しており使用不可能であった。また、１本だけ得られた単結晶のＧａＡｓ結晶を切断して転位密度を調べたところ、結晶のどの領域においても転位密度は５０００cm^{-2}を超えていた。

【００３５】

【発明の効果】本発明によれば、るつぼ底部の種結晶設置部内に種結晶を設置し、そのるつぼ内に化合物半導体の原料及び封止剤を入れ、そのるつぼを気密容器内に封入した後、該気密容器を縦型の加熱炉内に設置して前記原料及び前記封止剤をヒータにより加熱融解し、得られた原料融液を下側から徐々に冷却して前記種結晶から上方に向かって固化させることにより化合物半導体の単結晶を成長させるにあたって、前記るつぼとして、その底面がその中心に向かって徐々に低くなるように垂直方向に対して８０°以上９０°未満の所定角度をなして傾斜したるつぼを用いるとともに、結晶成長時に少なくともその傾斜したるつぼ底部分の結晶成長方向の温度勾配を１℃／cm以上５℃／cm未満となるように制御して結晶成長を行うようにしたため、結晶の増径部が形成されずに結晶育成開始後すぐに直胴部の育成が開始されるので、得られた単結晶インゴットからのウエハーの収率が高く生産性がよい。また、原料融液の固化時の温度勾配が小さく、温度ゆらぎが小さいため、双晶及び多結晶の発生が抑制され、高い歩留まりで単結晶が得られる。さらに、加熱炉は冷却媒体用の配管等が不要であるため、従来の加熱炉をそのまま使うことができるので、コストの増加を招くことなく、高品質の単結晶が高歩留まりで得られる。

【００３６】また、本発明によれば、前記るつぼの底面と側面との境界部分の曲率半径が０mm以上１０mm以下であるため、育成結晶の、結晶肩部から直胴部への移行部分の結晶成長時間が短くなり、ファセットの発生が抑制され、双晶の発生が防止される。

【図面の簡単な説明】

【図１】本発明の実施に使用されるるつぼの一例の断面図である。

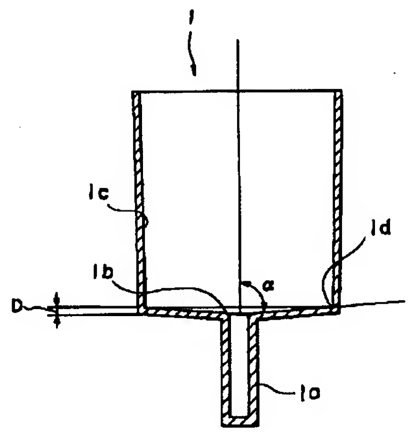
【図２】本発明をＶＧＦ法に適用した際に使用される結晶成長炉の概略図である。

【符号の説明】

１　るつぼ

１ａ　種結晶設置部
１ｂ　るつぼの底面
１ｃ　るつぼの側面
１ｄ　るつぼの角部
２　種結晶
３　原料
４　封止剤
５　気密容器
８　加熱炉（結晶成長炉）
９　ヒータ
１０　単結晶

【図１】



【図２】